

Subpart LLL—Standards of Performance for Onshore Natural Gas Processing: SO₂ Emissions

SOURCE: 50 FR 40160, Oct. 1, 1985, unless otherwise noted.

§ 60.640 Applicability and designation of affected facilities.

(a) The provisions of this subpart are applicable to the following affected facilities that process natural gas: each sweetening unit, and each sweetening unit followed by a sulfur recovery unit.

(b) Facilities that have a design capacity less than 2 long tons per day (LT/D) of hydrogen sulfide (H₂S) in the acid gas (expressed as sulfur) are required to comply with § 60.647(c) but are not required to comply with §§ 60.642 through 60.646.

(c) The provisions of this subpart are applicable to facilities located on land and include facilities located onshore which process natural gas produced from either onshore or offshore wells.

(d) The provisions of this subpart apply to each affected facility identified in paragraph (a) of this section which commences construction or modification after January 20, 1984.

(e) The provisions of this subpart do not apply to sweetening facilities producing acid gas that is completely reinjected into oil-or-gas-bearing geologic strata or that is otherwise not released to the atmosphere.

§ 60.641 Definitions.

All terms used in this subpart not defined below are given the meaning in the Act and in subpart A of this part.

Acid gas means a gas stream of hydrogen sulfide (H₂S) and carbon dioxide (CO₂) that has been separated from sour natural gas by a sweetening unit.

Natural gas means a naturally occurring mixture of hydrocarbon and nonhydrocarbon gases found in geologic formations beneath the earth's surface. The principal hydrocarbon constituent is methane.

Onshore means all facilities except those that are located in the territorial seas or on the outercontinental shelf.

Reduced sulfur compounds means H₂S, carbonyl sulfide (COS), and carbon disulfide (CS₂).

Sulfur production rate means the rate of liquid sulfur accumulation from the sulfur recovery unit.

Sulfur recovery unit means a process device that recovers element sulfur from acid gas.

Sweetening unit means a process device that separates the H₂S and CO₂ contents from the sour natural gas stream.

Total SO₂ equivalents means the sum of volumetric or mass concentrations of the sulfur compounds obtained by adding the quantity existing as SO₂ to the quantity of SO₂ that would be obtained if all reduced sulfur compounds were converted to SO₂ (ppmv or kg/DSCM).

E=the sulfur emission rate expressed as elemental sulfur, kilograms per hour (kg/hr) rounded to one decimal place.

R=the sulfur emission reduction efficiency achieved in percent, carried to one decimal place.

S=the sulfur production rate in kilograms per hour (kg/hr) rounded to one decimal place.

X=the sulfur feed rate, i.e., the H₂S in the acid gas (expressed as sulfur) from the sweetening unit, expressed in long tons per day (LT/D) of sulfur rounded to one decimal place.

Y=the sulfur content of the acid gas from the sweetening unit, expressed as mole percent H₂S (dry basis) rounded to one decimal place.

Z=the minimum required sulfur dioxide (SO₂) emission reduction efficiency, expressed as percent carried to one decimal place. Z_i refers to the reduction efficiency required at the initial performance test. Z_c refers to the reduction efficiency required on a continuous basis after compliance with Z_i has been demonstrated.

§ 60.642 Standards for sulfur dioxide.

(a) During the initial performance test required by § 60.8(b), each owner or operator shall achieve at a minimum, an SO₂ emission reduction efficiency (Z_i) to be determined from Table 1 based on the sulfur feed rate (X) and the sulfur content of the acid gas (Y) of the affected facility.

(b) After demonstrating compliance with the provisions of paragraph (a) of this section, the owner or operator shall achieve at a minimum, an SO₂ emission reduction efficiency (Z_c) to be determined from Table 2 based on the sulfur feed rate (X) and the sulfur content of the acid gas (Y) of the affected facility.

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§ 60.643 Compliance provisions.

(a)(1) To determine compliance with the standards for sulfur dioxide specified in § 60.642(a), during the initial performance test as required by § 60.8, the minimum required sulfur dioxide emission reduction efficiency (Z) is compared to the emission reduction efficiency (R) achieved by the sulfur recovery technology.

(i) If $R \geq Z_i$, the affected facility is in compliance.

(ii) If $R < Z_i$, the affected facility is not in compliance.

(2) Following the initial determination of compliance as required by § 60.8, any subsequent compliance determinations that may be required by the Administrator would compare R to Z_c .

(b) The emission reduction efficiency (R) achieved by the sulfur reduction technology shall be determined using the procedures in § 60.644(c)(1).

[50 FR 40160, Oct. 1, 1985, as amended at 54 FR 6679, Feb. 14, 1989]

§ 60.644 Test methods and procedures.

(a) In conducting the performance tests required in § 60.8, the owner or operator shall use as reference methods and procedures the test methods in appendix A of this part or other methods and procedures as specified in this section, except as provided in paragraph § 60.8(b).

(b) During a performance test required by § 60.8, the owner or operator shall determine the minimum required reduction efficiencies (Z) of SO_2 emissions as required in § 60.642 (a) and (b) as follows:

(1) The average sulfur feed rate (X) shall be computed as follows:

$$X = K Q_a Y$$

where:

X=average sulfur feed rate, long ton/day.

Q_a =average volumetric flow rate of acid gas from sweetening unit, dscf/day.

Y=average H_2S concentration in acid gas feed from sweetening unit, percent by volume.

$K = (32 \text{ lb S/lb-mole}) / [(100\%)(385.36 \text{ dscf/lb-mole})(2240 \text{ lb/long ton})]$
 $= 3.707 \times 10^{-7}$

(2) The continuous readings from the process flowmeter shall be used to determine the average volumetric flow rate (Q_a) in dscf/day of the acid gas from the sweetening unit for each run.

(3) The Tutwiler procedure in § 60.648 or a chromatographic procedure following ASTM E-260 (incorporated by reference—see § 60.17) shall be used to determine the H_2S concentration in the acid gas feed from the sweetening unit. At least one sample per hour (at equally spaced intervals) shall be taken during each 4-hour run. The arithmetic mean of all samples shall be the average

H_2S concentration (Y) on a dry basis for the run. By multiplying the result from the Tutwiler procedure by 1.62×10^{-3} , the units gr/100 scf are converted to volume percent.

(4) Using the information from paragraphs (b) (1) and (3), Tables 1 and 2 shall be used to determine the required initial (Z_i) and continuous (Z_c) reduction efficiencies of SO_2 emissions.

(c) The owner or operator shall determine compliance with the SO_2 standards in § 60.642 (a) or (b) as follows:

(1) The emission reduction efficiency (R) achieved by the sulfur recovery technology shall be computed for each run using the following equation:

$$R = (100 S) / (S + E)$$

(2) The level indicators or manual soundings shall be used to measure the liquid sulfur accumulation rate in the product storage tanks. Readings taken at the beginning and end of each run, the tank geometry, sulfur density at the storage temperature, and sample duration shall be used to determine the sulfur production rate (S) in kg/hr for each run.

(3) The emission rate (E) of sulfur shall be computed for each run as follows:

$$E = C_e Q_{sd} / K$$

where:

C_e =concentration of sulfur equivalent (SO_2 +TRS), g/dscm.

Q_{sd} =volumetric flow rate of effluent gas, dscm/hr.

K=conversion factor, 1000 g/kg.

(4) The concentration (C_e) of sulfur equivalent shall be the sum of the SO_2 and TRS concentrations, after being converted to sulfur equivalents. For each run and each of the test methods specified in this paragraph (c) of this section, the sampling time shall be at least 4 hours. Method 1 shall be used to select the sampling site. The sampling point in the duct shall be at the centroid of the cross-section if the area is less than 5 m² (54 ft²) or at a point no closer to the walls than 1 m (39 in.) if the cross-sectional area is 5 m² or more, and the centroid is more than 1 m (39 in.) from the wall.

(i) Method 6 shall be used to determine the SO_2 concentration. Eight samples of 20 minutes each shall be taken at 30-minute intervals. The arithmetic average in mg/dscm shall be the concentration for the run. The concentration in mg/dscm shall be multiplied by 0.5 to convert the results to sulfur equivalent.

(ii) Method 15 shall be used to determine the TRS concentration from reduction-type devices or where the oxygen content of the effluent gas is less than 1.0 percent by volume. The sampling rate shall be at least 3 liters/min (0.1 ft³/min) to insure minimum residence time in the sample line.

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Sixteen samples shall be taken at 15-minute intervals. The arithmetic average of all the samples shall be the concentration for the run. The concentration in ppm TRS as H₂S shall be multiplied by 1.352×10^{-6} to convert the results to sulfur equivalent.

(iii) Method 16A shall be used to determine the TRS concentration from oxidation-type devices or where the oxygen content of the effluent gas is greater than 1.0 percent by volume. Eight samples of 20 minutes each shall be taken at 30-minute intervals. The arithmetic average shall be the concentration for the run. The concentration in ppm TRS as H₂S shall be multiplied by 1.352×10^{-6} to convert the results to sulfur equivalent.

(iv) Method 2 shall be used to determine the volumetric flow rate of the effluent gas. A velocity traverse shall be conducted at the beginning and end of each run. The arithmetic average of the two measurements shall be used to calculate the volumetric flow rate (Q_{sd}) for the run. For the determination of the effluent gas molecular weight, a single integrated sample over the 4-hour period may be taken and analyzed or grab samples at 1-hour intervals may be taken, analyzed, and averaged. For the moisture content, two samples of at least 0.10 dscm (0.35 dscf) and 10 minutes shall be taken at the beginning of the 4-hour run and near the end of the time period. The arithmetic average of the two runs shall be the moisture content for the run.

(d) To comply with § 60.646(d), the owner or operator shall obtain the information required by using the monitoring devices in paragraph (b) of (c) of this section.

[54 FR 6679, Feb. 14, 1989]

§ 60.645 [Reserved]

§ 60.646 Monitoring of emissions and operations.

(a) The owner or operator subject to the provisions of § 60.642 (a) or (b) shall install, calibrate, maintain, and operate monitoring devices or perform measurements to determine the following operations information on a daily basis:

(1) The accumulation of sulfur product over each 24-hour period: The monitoring method may incorporate the use of an instrument to measure and record the liquid sulfur production rate, or may be a procedure for measuring and recording the sulfur liquid levels in the storage tanks with a level indicator or by manual soundings, with subsequent calculation of the sulfur production rate based on the tank geometry, stored sulfur density, and elapsed time between readings. The method shall be designed to be accurate within ± 2 percent of the 24-hour sulfur accumulation.

(2) The H₂S concentration in the acid gas from the sweetening unit for each 24-hour period: At least one sample per 24-hour period shall be collected and analyzed using the method specified in § 60.644(b)(1). The Administrator may require the owner or operator to demonstrate that the H₂S concentration obtained from one or more samples over a 24-hour period is within ± 20 percent of the average of 12 samples collected at equally spaced intervals during the 24-hour period. In instances where the H₂S concentration of a single sample is not within ± 20 percent of the average of the 12 equally spaced samples, the Administrator may require a more frequent sampling schedule.

(3) The average acid gas flow rate from the sweetening unit: The owner or operator shall install and operate a monitoring device to continuously measure the flow rate of acid gas. The monitoring device reading shall be recorded at least once per hour during each 24-hour period. The average acid gas flow rate shall be computed from the individual readings.

(4) The sulfur feed rate (X): For each 24-hour period, X shall be computed using the equation in § 60.644(b)(3).

(5) The required sulfur dioxide emission reduction efficiency for the 24-hour period: The sulfur feed rate and the H₂S concentration in the acid gas for the 24-hour period as applicable, shall be used to determine the required reduction efficiency in accordance with the provisions of § 60.642(b).

(b) Where compliance is achieved through the use of an oxidation control system or a reduction control system followed by a continually operated incineration device, the owner or operator shall install, calibrate, maintain, and operate monitoring devices and continuous emission monitors as follows:

(1) A continuous monitoring system to measure the total sulfur emission rate (E) of SO₂ in the gases discharged to the atmosphere. The SO₂ emission rate shall be expressed in terms of equivalent sulfur mass flow rates (kg/hr). The span of this monitoring system shall be set so that the equivalent emission limit of § 60.642(b) will be between 30 percent and 70 percent of the measurement range of the instrument system.

(2) Except as provided in paragraph (b)(3) of this section: A monitoring device to measure the temperature of the gas leaving the combustion zone of the incinerator, if compliance with § 60.642(a) is achieved through the use of an oxidation control system or a reduction control system followed by a continually operated incineration device. The monitoring device shall be certified by the manufacturer to be accurate to within ± 1 percent of the temperature being measured.

When performance tests are conducted under the provision of § 60.8 to demonstrate compliance

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with the standards under § 60.642, the temperature of the gas leaving the incinerator combustion zone shall be determined using the monitoring device. If the volumetric ratio of sulfur dioxide to sulfur dioxide plus total reduced sulfur (expressed as SO₂) in the gas leaving the incinerator is ≤0.98, then temperature monitoring may be used to demonstrate that sulfur dioxide emission monitoring is sufficient to determine total sulfur emissions. At all times during the operation of the facility, the owner or operator shall maintain the average temperature of the gas leaving the combustion zone of the incinerator at or above the appropriate level determined during the most recent performance test to ensure the sulfur compound oxidation criteria are met. Operation at lower average temperatures may be considered by the Administrator to be unacceptable operation and maintenance of the affected facility. The owner or operator may request that the minimum incinerator temperature be reestablished by conducting new performance tests under § 60.8.

(3) Upon promulgation of a performance specification of continuous monitoring systems for total reduced sulfur compounds at sulfur recovery plants, the owner or operator may, as an alternative to paragraph (b)(2) of this section, install, calibrate, maintain, and operate a continuous emission monitoring system for total reduced sulfur compounds as required in paragraph (d) of this section in addition to a sulfur dioxide emission monitoring system. The sum of the equivalent sulfur mass emission rates from the two monitoring systems shall be used to compute the total sulfur emission rate (E).

(c) Where compliance is achieved through the use of a reduction control system not followed by a continually operated incineration device, the owner or operator shall install, calibrate, maintain, and operate a continuous monitoring system to measure the emission rate of reduced sulfur compounds as SO₂ equivalent in the gases discharged to the atmosphere. The SO₂ equivalent compound emission rate shall be expressed in terms of equivalent sulfur mass flow rates (kg/hr). The span of this monitoring system shall be set so that the equivalent emission limit of § 60.642(b) will be between 30 and 70 percent of the measurement range of the system. This requirement becomes effective upon promulgation of a performance specification for continuous monitoring systems for total reduced sulfur compounds at sulfur recovery plants.

(d) For those sources required to comply with paragraph (b) or (c) of this section, the average sulfur emission reduction efficiency achieved (R) shall be calculated for each 24-hour clock interval. The 24-hour interval may begin and end at any selected clock time, but must be consistent. The 24-

hour average reduction efficiency (R) shall be computed based on the 24-hour average sulfur production rate (S) and sulfur emission rate (E), using the equation in § 60.644(c)(1).

(1) Data obtained from the sulfur production rate monitoring device specified in paragraph (a) of this section shall be used to determine S.

(2) Data obtained from the sulfur emission rate monitoring systems specified in paragraphs (b) or (c) of this section shall be used to calculate a 24-hour average for the sulfur emission rate (E). The monitoring system must provide at least one data point in each successive 15-minute interval. At least two data points must be used to calculate each 1-hour average. A minimum of 18 1-hour averages must be used to compute each 24-hour average.

(e) In lieu of complying with (b) or (c) of this section, those sources with a design capacity of less than 150 LT/D of H₂S expressed as sulfur may calculate the sulfur emission reduction efficiency achieved for each 24-hour period by:

$$R = \frac{0.0236 S}{X} \quad (100 \text{ percent})$$

Where:

R=the sulfur dioxide removal efficiency achieved during the 24-hour period, percent;

S=the sulfur production rate during the 24-hour period, kg/hr;

X=the sulfur feed rate in the acid gas, LT/D; and
0.0236=conversion factor, LT/D per kg/hr.

(f) The monitoring devices required in paragraphs (b)(1), (b)(3) and (c) of this section shall be calibrated at least annually according to the manufacturer's specifications, as required by § 60.13(b).

(g) The continuous emission monitoring systems required in paragraphs (b)(1), (b)(3), and (c) of this section shall be subject to the emission monitoring requirements of § 60.13 of the General Provisions. For conducting the continuous emission monitoring system performance evaluation required by § 60.13(c), Performance Specification 2 shall apply, and Method 6 shall be used for systems required by paragraph (b) of this section.

[50 FR 40160, Oct. 1, 1985, as amended at 54 FR 6680, Feb. 14, 1989]

§ 60.647 Recordkeeping and reporting requirements.

(a) Records of the calculations and measurements required in § 60.642 (a) and (b) and § 60.646 (a) through (g) must be retained for at least 2 years following the date of the measurements by owners and operators subject to this subpart. This requirement is included under § 60.7(d) of the General Provisions.

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(b) Each owner or operator shall submit a written report of excess emissions to the Administrator semiannually. For the purpose of these reports, excess emissions are defined as:

(1) Any 24-hour period (at consistent intervals) during which the average sulfur emission reduction efficiency (R) is less than the minimum required efficiency (Z).

(2) For any affected facility electing to comply with the provisions of § 60.646(b)(2), any 24-hour period during which the average temperature of the gases leaving the combustion zone of an incinerator is less than the appropriate operating temperature as determined during the most recent performance test in accordance with the provisions of § 60.646(b)(2). Each 24-hour period must consist of at least 96 temperature measurements equally spaced over the 24 hours.

(c) To certify that a facility is exempt from the control requirements of these standards, each owner or operator of a facility with a design capacity less than 2 LT/D of H₂S in the acid gas (expressed as sulfur) shall keep, for the life of the facility, an analysis demonstrating that the facility's design capacity is less than 2 LT/D of H₂S expressed as sulfur.

(d) Each owner or operator who elects to comply with § 60.646(e) shall keep, for the life of the facility, a record demonstrating that the facility's design capacity is less than 150 LT/D of H₂S expressed as sulfur.

(e) The requirements of paragraph (b) of this section remain in force until and unless EPA, in delegating enforcement authority to a State under section 111(c) of the Act, approves reporting requirements or an alternative means of compliance surveillance adopted by such State. In that event, affected sources within the State will be relieved of obligation to comply with paragraph (b) of this section, provided that they comply with the requirements established by the State.

§ 60.648 Optional procedure for measuring hydrogen sulfide in acid gas—Tutwiler Procedure.¹

(a) When an instantaneous sample is desired and H₂S concentration is ten grains per 1000 cubic foot or more, a 100 ml Tutwiler burette is used. For concentrations less than ten grains, a 500 ml Tutwiler burette and more dilute solutions are used. In principle, this method consists of titrating hydrogen sulfide in a gas sample directly with a standard solution of iodine.

(b) *Apparatus.* (See Figure 1.) A 100 or 500 ml capacity Tutwiler burette, with two-way glass

stopcock at bottom and three-way stopcock at top which connect either with inlet tubulature or glass-stoppered cylinder, 10 ml capacity, graduated in 0.1 ml subdivision; rubber tubing connecting burette with leveling bottle.

(c) *Reagents.* (1) Iodine stock solution, 0.1N. Weight 12.7 g iodine, and 20 to 25 g cp potassium iodide for each liter of solution. Dissolve KI in as little water as necessary; dissolve iodine in concentrated KI solution, make up to proper volume, and store in glass-stoppered brown glass bottle.

(2) Standard iodine solution, 1 ml=0.001771 g I. Transfer 33.7 ml of above 0.1N stock solution into a 250 ml volumetric flask; add water to mark and mix well. Then, for 100 ml sample of gas, 1 ml of standard iodine solution is equivalent to 100 grains H₂S per cubic foot of gas.

(3) Starch solution. Rub into a thin paste about one teaspoonful of wheat starch with a little water; pour into about a pint of boiling water; stir; let cool and decant off clear solution. Make fresh solution every few days.

(d) *Procedure.* Fill leveling bulb with starch solution. Raise (L), open cock (G), open (F) to (A), and close (F) when solutions starts to run out of gas inlet. Close (G). Purge gas sampling line and connect with (A). Lower (L) and open (F) and (G). When liquid level is several ml past the 100 ml mark, close (G) and (F), and disconnect sampling tube. Open (G) and bring starch solution to 100 ml mark by raising (L); then close (G). Open (F) momentarily, to bring gas in burette to atmospheric pressure, and close (F). Open (G), bring liquid level down to 10 ml mark by lowering (L). Close (G), clamp rubber tubing near (E) and disconnect it from burette. Rinse graduated cylinder with a standard iodine solution (0.00171 g I per ml); fill cylinder and record reading. Introduce successive small amounts of iodine thru (F); shake well after each addition; continue until a faint permanent blue color is obtained. Record reading; subtract from previous reading, and call difference D.

(e) With every fresh stock of starch solution perform a blank test as follows: introduce fresh starch solution into burette up to 100 ml mark. Close (F) and (G). Lower (L) and open (G). When liquid level reaches the 10 ml mark, close (G). With air in burette, titrate as during a test and up to same end point. Call ml of iodine used C. Then,

Grains H₂S per 100 cubic foot of gas=100 (D—C)

(f) Greater sensitivity can be attained if a 500 ml capacity Tutwiler burette is used with a more dilute (0.001N) iodine solution. Concentrations less than 1.0 grains per 100 cubic foot can be determined in this way. Usually, the starch-iodine end point is much less distinct, and a blank deter-

¹ Gas Engineers Handbook, Fuel Gas Engineering Practices, The Industrial Press, 93 Worth Street, New York, NY, 1966, First Edition, Second Printing, page 6/25 (Docket A-80-20-A, Entry II-1-67).

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mination of end point, with H₂S-free gas or air, is required.

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Figure 1. Tutwiler burette (lettered items mentioned in text).